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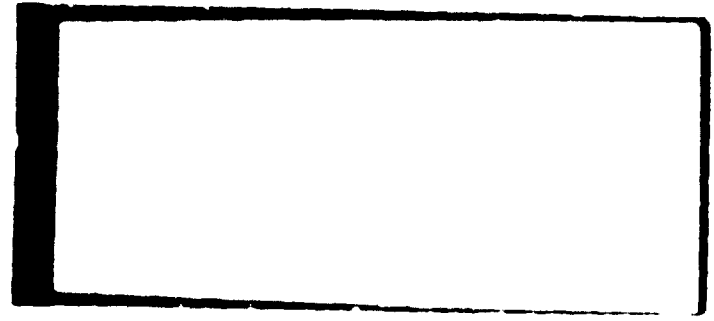
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SPECIAL REPORT
DESTRUCTIVE LEAK TEST COMPARISON
FOR TWO AVAILABLE MC-6/U BASES

PER PER CONTRACT MODIFICATION NO. 3
CONTRACT NO. DA-36-099-SC-01271
ORDER NO. 7610-PP-59-81-81

PLACED BY: USAEMA
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I. INTRODUCTION

This special test report "Destructive Leak Test Comparison for Two Types of Available HC-6/U Bases" is submitted by Bulova to USAF as a supplement to the "Final Report, PEM on Type CR-(XM-27)/U Crystal Units" (Contract No. D4-SC-36-039-SC-81271 Order No. 7618-PP-59-81-81). The test report is submitted as consideration for modification of the PEM Contract noted above.

Destructive leak tests were performed on 400 crystal units consisting of 200 units fabricated with each of two HC-6/U base designs. The tests are of general interest since many MIL-type crystal units are housed in HC-6/U holders although the tests have no direct application to CR-(XM-27)/U Crystal Units produced on the PEM Contract. The tests were performed on completed crystal units for missile application on another Contract.

In this Report, two destructive test conditions are described: accelerated thermal shock tests and HC-6/U terminal-pin bending tests. In these tests, 100 crystal units of each of the two HC-6/U Base designs were allocated to each of the two destructive tests. For measuring resulting leak rates, a Veeco (Model MS-9ABC) Mass Spectrometer Leak Detector was employed; crystal units exhibiting leak rates exceeding 10^{-6} cc/s were classified as rejects.

In the included test results, a comparison of cumulative leak rejects indicates significant differences in survival capability for the two HC-6/U base designs which were tested. The more rugged HC-6/U base type can be advantageously employed for low frequency crystals; at higher frequencies, it has limited application because adverse holder inductance and holder resistance effects are introduced.

A. CRYSTALS EMPLOYED IN TESTS

A total of 400 DT Cut crystal units at approximately 200 KC were identically mounted on HC-6/U bases containing crystal mounting structures which were designed to survive high shock and high frequency vibration impacts with no significant change in electrical performance. The crystal units were designed for operation at $75^{\circ}\text{C} \pm 5^{\circ}\text{C}$. HC-6/U covers were attached by soldering to the HC-6/U bases and the "breather" holes were sealed with solder after 2 prior evacuation cycles followed by "back filling" with helium gas under a Bell jar. Of the 400 crystal units, 200 crystal units contained Type I HC-6/U bases and 200 crystal units contained Type II HC-6/U bases. The 2 base types are differentiated in B. below.

B. MATERIAL COMPOSITION FOR THE 2 HC-6/U BASE TYPES

<u>Part Description</u>	<u>Type I</u>	<u>Type II</u>
2 terminal pins	Kovar #920050 or equal	52% Nickel 48% Iron Alloy
Base (Metal)	Kovar #910010 or equal	Steel, cold-rolled strip condition No. 3 per Specification QQ-S-00640
Glass Bead	Corning No. 705-2 Glass	Corning No. 9010 or equal

After assembling (both Type I and Type II), the base assembly shall be hot-tin-dipped or electro-tin-plated in accordance with type I finish of MIL-F-14072.

The HC-6/U cover (both Type I and Type II) shall be copper-nickel-sinc alloy, composition No. 5, annealed soft temper, per Specification QQ-C-585.

Note: Type I HC-6/U base is commonly referred to as the "Kovar" base and Type II as the "compression seal" base.

C. ACCELERATED THERMAL SHOCK TEST PROCEDURE

Permanent changes in operating characteristics and physical damage produced during temperature cycling result principally from variations in dimensions and other physical properties, and from alternate condensation and freezing of atmospheric moisture. Effects of temperature cycling include cracking and delamination of finishes, embedding compounds and other materials, opening of terminal seals and case seams, and changes in electrical characteristics due to mechanical displacement or rupture of conductors or of insulating materials. When temperature cycling is accelerated, these effects are more pronounced and the testing may be termed "destructive."

In each accelerated thermal shock test cycle, the crystal units were alternately exposed to temperature end points of -40°C and 100°C with a half hour total exposure at each end point for a total period of 16 hours beginning at 8:00 AM and ending at midnight. At the conclusion of temperature cycling at midnight, the crystal units remained in the 100°C temperature chamber until the next working day (of a 5 day work-week) when leak tests were initiated at approximately 9:00 A.M. The crystal units were alternately temperature cycled and leak tested for 28 working days ($5\frac{1}{2}$ weeks) consisting of 14 temperature cycling tests (14 sixteen hour days) and 14 leak test days. For the Leak Tests, a Veeco (Model No. MS-9ABC) Mass Spectrometer Leak Detector was employed; the HC-6/U crystal enclosures were "back-filled" with helium gas to provide the leak detection medium.

Temperature Cycling Test Procedure

1. For temperature control, two Midwest Box Temperature Test Chambers are employed. One test chamber is stabilized at $-40^{\circ}\text{C} \pm 5^{\circ}\text{C}$; the other chamber is stabilized at $100^{\circ}\text{C} \pm 3^{\circ}\text{C}$.
2. Prior to initiating the test cycle, individually measure and record the series frequency and series resistance of all crystal units under test at $75^{\circ}\text{C} \pm 1^{\circ}\text{C}$; a Single Crystal Oven shall be employed for temperature regulation. At the start of the first test cycle, the test lot consisted of 200 crystal units of which 100 units were fabricated with Type II HC-6/U bases (See Section II.B) and 100 units with Type II bases.
3. Upon completion of step 2. above, pin insert the crystal units into aluminum handling racks, (25 crystal unit capacity) and place the rack assemblies into the -40°C test chamber for a period of 30 minutes.
4. Remove the rack assemblies (with crystals) and immediately place the rack assemblies into the 100°C test chamber for a period of 30 minutes. Crystal transfer from one chamber to the other shall be accomplished in 5 to 10 seconds.

5. Steps 3. and 4. above are repeated 16 times for a total test time of 16 hours.
6. At the conclusion of step 5., the crystals remain in the 100°C test chamber until leak testing is initiated on the following working day.
7. Steps 1 through 6 are repeated for a total of 14 times (14 sixteen hour days) alternated with 14 days of leak testing except that crystal units exhibiting leak rates exceeding 10^{-6} cc/s (in subsequent leak tests) are eliminated from further temperature cycling.

D. LEAK TEST PROCEDURE

A Veeco (Model No. MS-9ABC) Mass Spectrometer Leak Detector was employed in the Leak Tests. The heart of the Leak Detector is the "Vee Tube" mass spectrometer. As a sensing element, the "Vee Tube" detects helium gas and produces an electrical signal in proportion to the rate of flow of helium; it is capable of measuring leak rates down to 10^{-10} cc/s. The "Vee Tube" may be divided into 3 sections, each of which performs separate functions, but all contributing to the detection of very small quantities of helium. A description of the 3 sections and functions follows:

1. Ion Gun: In the Ion Gun, a tungsten filament, heated white-hot by current, produces an electron beam; the electron beam bombards escaping gas molecules from the test sample and converts the molecules to positive ions. A repeller grid, operated at a slight positive potential, repels the positive gas ions down through a slit to form a beam.
2. Alnico VI Magnet: The magnet separates helium ions from all other ions by separating the beam into a spectrum of separate beams, each of which contains only ions of the same mass.
3. Ion Collector: After helium ions have traversed the lower end of the "Vee Tube", they strike the target or ion collector. The flow of positively charged ions to the collector is called the "ion current." The current is detected and amplified by a special electrometer tube which is mounted within the spectrometer tube itself. The electrometer tube is connected to the external amplifier by means of a cable. The amplified helium current appears as a visual indication on the portable Leak Indicator Meter.

Since the "Vee Tube" operates in a high vacuum, a complete high vacuum system (with Bell Jar) is provided, consisting of special pumps, gauges and valves. To achieve the high vacuum, a 3 stage pumping cycle is employed. After preliminary pumping with a roughing pump, a forepump is engaged to achieve a vacuum pressure down to 20 microns (minimum). In the last stage of pumping, a diffusion pump is engaged (at 100 Micron maximum) to achieve a pressure down to 10^{-3} micron (minimum). A liquid nitrogen cold trap aids in achieving these low pressures.

Other auxiliary components of the Leak Detector serve to either maintain proper operating conditions within the "Vee Tube" or to measure its electronic output.

The Pressure-Vacuum test method was employed on all crystal units under Leak Tests. Crystal units in HC-6/U holders having an internal helium pressure were placed inside a vacuum chamber (under a Bell Jar) which is connected to the "Vee Tube" where escaping helium gas will be detected.

Prior to Leak Testing, Leak Indicator Meter readings are calibrated by comparison with a Known Leak Rate Standard. Actual Leak Indicator Meter readings are relative since they are influenced by the background helium count in the atmosphere, contamination in the vacuum system and other causes. To compensate for cumulative rate increases resulting from these effects, correction factors are introduced in leak rate formulas. From these correlated readings, a Leak Indicating Meter reading corresponding to a leak rate of 10^{-6} cc/s was established as a reject point; crystal units with Meter readings exceeding the established figure are classified as rejects.

For operating instructions, see "Operation and Maintenance Manual" for the Veeco Mass Spectrometer Leak Detector for Model MS-9ABC by Vacuum Electronics Corp., Terminal Drive, Plainville, Long Island, N.Y.

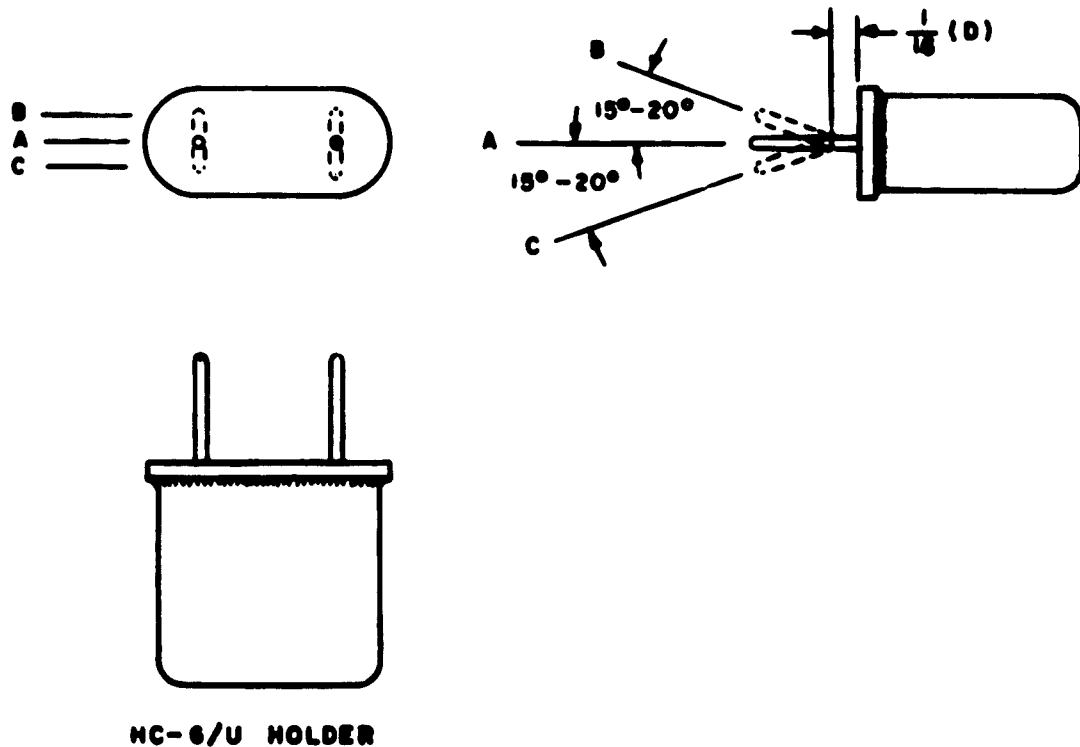
Leak Test Procedure

1. Measure the series frequency and resistance at $75^{\circ}\text{C} \pm 1^{\circ}\text{C}$ of all crystal units under test; a single crystal oven shall be employed for temperature regulation. At the start of the first leak test, the test lot consisted of 400 crystal units of which 200 units were fabricated with Type I HC-6/U bases and 200 crystal units were fabricated with Type II HC-6/U bases (See Section II.B). Of 200 crystal units of each HC-6/U base type, 100 units were subjected to Accelerated Thermal Shock Tests and 100 were subjected to Terminal Pin Bending Tests (See Section II. E following).
2. To maintain identity, all crystal units have been assigned serial numbers. In addition, the crystal units have been separated both as to base type and type of test (thermal or terminal pin bending tests) to which they have been subjected. All crystal units with the same HC-6/U base type are further sub-divided into sub-groups consisting of 10 crystal units each.
3. A sub-group (10 crystal units) is placed in the Vacuum chamber of the Veeco Leak Detector unit; the chamber is evacuated to a pressure below 10^{-1} microns. The "Vee Tube" is energized and the leak rate is measured. If the combined leak rate (for the 10 crystal units under test) does not exceed 10^{-6} cc/s on the correlated Leak Indicator Meter reading, the 10 crystal units are ACCEPTED in the leak test.

4. If the leak rate in 3. above exceeds 10^{-6} cc/s, remove the 10 crystal units from the vacuum chamber and separate the 10 crystal units into 2 groups consisting of 5 crystal units per group.
5. Continue to sub-divide into smaller lots down to individual crystal testing where necessary to "weed out" all crystal units with a leak rate exceeding 10^{-6} cc/s. Individual crystal units exceeding 10^{-6} cc/s are classified as REJECTS and are eliminated from further tests.
6. Repeat steps 3. through 5. above on all remaining crystals.
7. Crystal units which have been ACCEPTED in the leak test are placed into a 100°C circulating oven for storage until the thermal shock tests or terminal pin bending tests (as appropriate) are resumed.

E. TERMINAL PIN-BENDING TESTS

The sketch below depicts the procedure employed in the Terminal Pin-Bending Tests on the HC-6/U bases.



In ONE BENDING CYCLE (manually with long-nose pliers), the 2 pins are individually bent from position A to B to C and back to A from a point (D) approximately 1/16" from the junction of the terminal pin with the Corning glass bead. This test simulates, in exaggerated degree, mechanical pin-displacements that may occur through the use of mis-aligned crystal sockets, through careless crystal handling by an operator or by inadvertant dropping.

In the test, we introduce deliberate mechanical stresses with resultant strains to the Corning glass bead with a view of comparing survival capability for Type I versus Type II HC-6/U base. In assessing damage to the Corning glass bead, the crystal units were subjected to a subsequent leak test. Crystal units with a leak rate exceeding 10^{-6} cc/s were classified as REJECTS and were eliminated from any further tests. Crystal units which were ACCEPTED in subsequent leak tests were subjected to additional Terminal Pin Bending Cycles alternated with leak tests. In time intervals between leak and pin bending tests, the crystal units are stored in circulating ovens maintained at $100^{\circ}\text{C} \pm 3^{\circ}\text{C}$.

A total of 200 crystal units consisting of 100 units each fabricated with Type I and Type II HC-6/U bases were subjected to Terminal Pin Bending Tests. Of the 100 units fabricated with the Type II base, 50 units were coated with an epoxy formulation on the glass bead around the terminal pins; the other 50 units were not coated. The formulation consisted of a 2 part (A and B) thermosetting epoxy "Scotchcast" Brand Resin No. 5; for application, 2 parts A were mixed with 1 part B and applied with a Q tip or eye-dropper to the exposed glass bead portion around the terminal pins of the Type II HC-6/U base. The epoxy coated units are placed in a 100°C oven for a period of 1 hour. The units are removed from the oven and the epoxy "hardens" after a 2 hour exposure to room temperature air.

F. CRYSTAL RESISTANCE CHANGE VS. LEAK RATE

It is commonly known that mounted crystals in evacuated enclosures exhibit lower series resistance values and higher Q's than comparable crystals in "gas-loaded" enclosures. Crystals in metal enclosures such as the HC-6/U are normally "back-filled" with a selected inert gas prior to sealing. Of all inert gases, helium presents a minimum "gas loading" effect since it has the lowest atomic weight ("gas loading" or vibration suppression increases with atomic weight). By employing helium as the "back fill" gas (at atmospheric pressure) for these tests, we accomplish the following:

1. The use of helium permits rapid and accurate determination of leak rates when used in conjunction with the Veeco Mass Spectrometer Leak Detector Unit since the unit works on the principle of flow rate rather than accumulation techniques (e.g. the Radiflo Leak Detector).
2. By employing helium, resistance in the tests is an indirect indication of leaks. All recorded measurements of crystal series resistance were made at the operating temperature of $75^{\circ}\text{C} \pm 1^{\circ}\text{C}$; the 200 crystal units (100 each of Type I and Type II HC-6/U bases) in the resistance variation tests were the same 200 crystal units which were subjected to the Thermal Shock Test. In many respects, the resistance variation test was more sensitive than the Veeco Mass Spectrometer Leak Detector test since some crystal units which passed the leak test exhibited resistance increases of 100 ohms or more. The tests indicate a significant difference in the ability of the 2 HC-6/U bases to retain helium gas (over a 6 week period).

III. TEST RESULTS

A condensed summary of test results appears in tabular and graphic form on page 9. In the graph numbered 1., the complete test lot consisted of 100 crystal units each fabricated with Type I and Type II HC-6/U bases. A comparison of initial crystal series resistance distributions (at 75°C) for the 2 HC-6/U base types indicates superior helium retention for Type II over Type I base. Since the crystals were identically processed, the result is significant. Resistance values in the curve apply to measurements made before subjecting the units to Thermal Shock Tests.

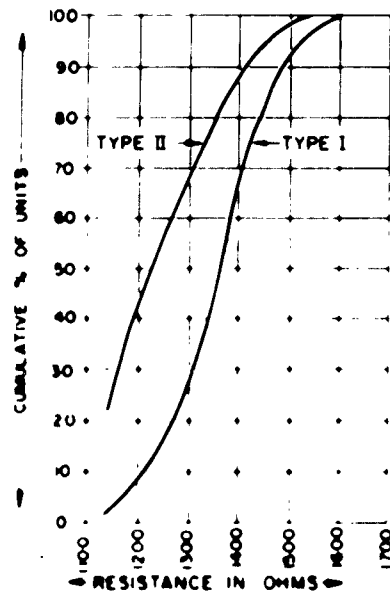
The graph numbered 2. represents crystal series resistance changes (at 75°C) after 14 Thermal Shock Test Cycles were applied. A comparison of test result distributions also indicates a marked difference in the ability of the 2 base types to survive accelerated Thermal Shock Tests without exhibiting major resistance changes (major helium losses). Type II HC-6/U bases produced superior results.

The tabulation numbered 3. is self-explanatory. It corroborates results in graphs 1. and 2. on the basis of the 17 leak Test Cycles which were performed.

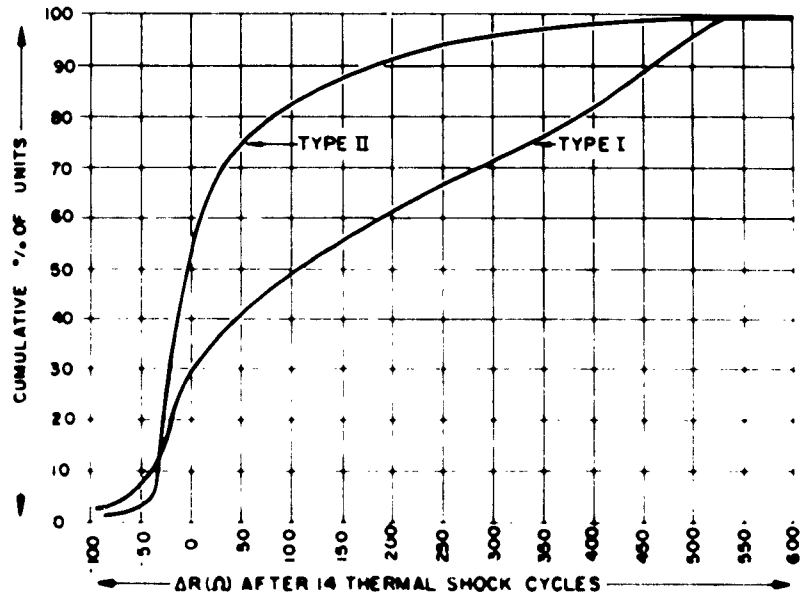
The tabulation numbered 4. represents results of terminal pin bending tests. A comparison of subsequent cumulative leak Test rejects for the 2 HC-6/U base types indicates superior test results for the Type II base.

CONDENSED SUMMARY OF TEST RESULTS

1. CRYSTAL SERIES RESISTANCE AT 75° C.
BEFORE THERMAL SHOCK TESTS



2. CRYSTAL SERIES RESISTANCE CHANGE AT 75° C
AFTER 14 THERMAL SHOCK TEST CYCLES



THERMAL SHOCK TEST CYCLES	NUMBER OF LEAKS		CUMULATIVE LEAKS		RELATIVE % SUBJECTS	
	TYPE I		TYPE II		TYPE I	
	NO. LEAKS	BASE	NO. LEAKS	BASE	NO. LEAKS	BASE
1	0	1	0	1	0%	0%
2	0	1	10	1	10%	0%
3	0	1	10	1	10%	0%
4	1	1	11	1	11%	0%
5	0	1	11	1	11%	0%
6	0	1	11	1	11%	0%
7	0	1	11	1	11%	0%
8	0	1	11	1	11%	0%
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93	0	1	11	1	11%	0%
94	0	1	11	1	11%	0%
95	0	1	11	1	11%	0%
96	0	1	11	1	11%	0%
97	0	1	11	1	11%	0%
98	0	1	11	1	11%	0%
99	0	1	11	1	11%	0%
100	0	1	11	1	11%	0%

REJECT POINT: LEAK RATE $\geq 10^{-6}$ cc/s

THERMAL SHOCK TEST CYCLES	NUMBER OF LEAKS		CUMULATIVE LEAKS		RELATIVE % SUBJECTS	
	TYPE I		TYPE II		TYPE I	
	NO. LEAKS	BASE	NO. LEAKS	BASE	NO. LEAKS	BASE
1	0	1	0	1	0%	0%
2	0	1	0	1	0%	0%
3	0	1	0	1	0%	0%
4	0	1	0	1	0%	0%
5	0	1	0	1	0%	0%
6	0	1	0	1	0%	0%
7	0	1	0	1	0%	0%
8	0	1	0	1	0%	0%
9	0	1	0	1	0%	0%
10	0	1	0	1	0%	0%
11	0	1	0	1	0%	0%
12	0	1	0	1	0%	0%
13	0	1	0	1	0%	0%
14	0	1	0	1	0%	0%
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73	0	1	0	1	0%	0%
74	0	1	0	1	0%	0%
75	0	1	0	1	0%	0%
76	0	1	0	1	0%	0%
77	0	1	0	1	0%	0%
78	0	1	0	1	0%	0%
79	0	1	0	1	0%	0%
80	0	1	0	1	0%	0%
81	0	1	0	1	0%	0%
82	0	1	0	1	0%	0%
83	0	1	0	1	0%	0%
84	0	1	0	1	0%	0%
85	0	1	0	1	0%	0%
86	0	1	0	1	0%	0%
87	0	1	0	1	0%	0%
88	0	1	0	1	0%	0%
89	0	1	0	1	0%	0%
90	0	1	0	1	0%	0%
91	0	1	0	1	0%	0%
92	0	1	0	1	0%	0%
93	0	1	0	1	0%	0%
94	0	1	0	1	0%	0%
95	0	1	0	1	0%	0%
96	0	1	0	1	0%	0%
97	0	1	0	1	0%	0%
98	0	1	0	1	0%	0%
99	0	1	0	1	0%	0%
100	0	1	0	1	0%	0%

TESTS DISCONTINUED AFTER SIXTH BONDING CYCLE BECAUSE TINS BEGAN TO RUPTURE AT HEAD POINT.

OF 22 TYPE II SUBJECTS, 17 WERE ON BASES WITH NO SPOUT IN THE GLASS HEAD.

IV. CONCLUSIONS

Leak Test results on crystal units subjected to either destructive Thermal Shock or Terminal Pin Bending Tests provide evidence that damage to the Corning No. 705-2 glass bead in Type I HC-6/U bases is more extensive than damage to Corning NO. 9010 glass bead in Type II bases. The addition of an Epoxy resin to the glass bead reduced the number of leak Test rejects on Type II bases to 29.4% of the un-coated counterpart bases (5/17).

Reduced to numbers, Type II HC-6/U bases exhibit these advantages over Type I bases:

1. Type II bases retain helium for a longer period of time. After 14 Thermal Shock Test Cycles, 82% of the crystal units with Type II bases increased in resistance less than 100 ohms; for comparison, only 49% of the crystal units with Type I bases exhibited a resistance increase of less than 100 ohms.
2. After 14 Thermal Shock Test Cycles, crystal units with Type II HC-6/U bases produced only 26.4% (14/53) of the total rejects produced with Type I bases.
3. After the Pin Bending Cycles, crystals fabricated with Type II HC-6/U bases (not coated with Epoxy) produced only 41.5% of the total number of leak Rejects exhibited by crystals with Type I bases. Epoxy coated Type II bases had only 12.2% as many leak Rejects as Type I bases.